A novel Microwave Frequency Scanning Capacitance Microscope

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Abstract

We report a new technique of scanning capacitance microscopy at microwave frequencies. A near field scanning microwave microscope probe is kept at a constant height of about 1 nm above the sample with the help of Scanning Tunneling Microscope (STM) feedback. The microwaves are incident onto the sample through a coaxial resonator that is terminated at one end with a sharp tip (the same tip is used to conduct STM), and capacitively coupled to a feedback circuit and microwave source at the other end. The feedback circuit keeps the source locked onto the resonance frequency of the resonator and outputs the frequency shift and quality factor change due to property variations of the sample. The spatial resolution due to capacitance variations is ≤ 2.5 nm. The microscope is broadband and experiments were performed from 7 GHz to 11 GHz. We develop a quantitative transmission line model that treats the tip to sample interaction as a series combination of capacitance and effective sheet resistance in the sample.

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Introduction: Scanning Capacitance Microscopy can be used to map spatial variations of the topography of conducting materials, or the dielectric properites of thin films and bulk insulators[1–3]. In general, such microscopes detect a change in capacitance, δC, by means of a resonant circuit that includes the probe-sample capacitance. An early version of such a microscope employed a diamond stylus in contact with the sample. When the stylus was touching the sample, a probe electrode (attached to the stylus) was 20nm above the sample. The electrode to sample capacitance was measured through the changing resonant frequency of an inductor/capacitor (LC) resonant circuit[1]. With this microscope the lateral resolution claimed was 100nm and vertical resolution of 0.3nm. Other capacitance microscopes have been made by adding a similar resonant capacitance sensor to an Atomic Force Microscope[2] and a Scanning Tunneling Microscope[3](STM). The lateral topographic resolutions reported were 75nm and 25nm, respectively. These microscopes have been used for dopant profiling in semiconductors[4–6], for example.

However, in many materials of interest, measurement of loss is crucial to extract the interesting physics. For example, certain colossal magneto-resistance (CMR) materials have "metallic" and "insulating" phases that coexist on very small (almost 1-2 nm) length scales[7]. In thin films of $Bi_2Sr_2CaCu_2O_{8+\delta}$, STM spectroscopic data shows evidence for coexisting superconducting and semiconducting phases[8] on similar length scales. One way to distinguish between the two phases is to locally measure ohmic losses. Existing capacitance microscopes are not designed to image such quantities. A near-field microwave microscope would be the right tool, and can be utilized to quantitatively extract losses in the form of sheet resistance (R_x). However, sub-micron spatial resolution is required to distinguish the finely intermixed phases.

Our objective now is to obtain high-resolution loss imaging, in conjunction with capacitance microscopy. The quantitative sheet resistance (R_x) of a sample was extracted earlier[9], from the frequency shift and quality factor of the microscope resonator with lateral resolution in the 10's to 100's of μm. We now want to improve the spatial resolution, while maintaining the sensitive loss imaging capability.

With a scanning microwave microscope, one can illuminate a controlled localized area with microwave fields and currents. In the past, some near-field microwave microscopes have utilized STM[10,11] or AFM tips[12–14]simply to concentrate rf electric field on a sample. The sharp end of the tip acts like a "lightening rod", enhancing the spatial resolution for microwave microscopy. In an earlier version of our microwave microscope, an STM tip was also used to focus electric fields on the surface[15]. Because the tip was in contact with the sample, with contact force of about 60 μN, the best spatial resolution achieved was $\simeq 1$ μm. The contact force flattened the imaging end of the tip, increasing the radius of curvature. One way to improve the spatial resolution of the microscope is to prevent the tip from touching the sample. To achieve this, the new version of the near-field microwave microscope (Fig. 1) has STM feedback integrated to maintain a roughly 1 nm constant height during scanning.

Experiment: The microscope, schematically presented in Fig. 1 is similar to the versions discussed at length in prior publications[15–19]. Changes made to our microscope include using a bias tee to make the DC connection to STM feedback while maintaining an AC coupling to the microwave source and feedback circuit. Also, the sample is now on a XYZ piezoelectric (piezo) translation stage, instead of an XY motor stage. At one end of the coaxial resonator is an open ended coaxial probe with a sharp STM tip sticking out of its center conductor. As with our previous microscopes[15], the other end of the resonator is capacitively coupled to a microwave source and a feedback circuit via a directional coupler and a diode detector, as shown in Fig.1. The feedback circuit keeps the source locked onto the resonance frequency f_0, of the resonator and it gives the frequency shift (Δf) and quality factor (Q) as output signals. However, the probe-sample seperation is maintained by a constant current STM feedback loop during scanning.

Many interesting materials have transition temperatures well below room temperature, requiring a cryogenic microscope. We use a commercially available Oxford Cryostat to cool the sample and part of the microscope. The sample can be cooled to any temperature between 4.2K and room temperature. The quality factor of the microscope is enhanced at

low temperatures due to the decrease of microwave losses in the resonator.

Model: The inset of figure 1 shows a closer look at the tip to sample interaction. This interaction is modelled as an effective capacitance C_x in series with the losses in the conducting sample due to ohmic dissipation, R_x. The complex load impedance presented to the microscope is $Z_x = R_x + (1/i\omega C_x)$.

Our quantitative understanding of the microscope is based on a transmission line model developed earlier[16,17]. In this model, the frequency shift and Q of the microscope depend both on C_x and R_x. In the region of interest, an estimate (discussed below) for the capacitance at a height of 1 nm is $C_x \simeq 10$ fF, giving a capacitive reactance ($\mathrm{Im}[Z_x]$) on the order of 2 kΩ at 7.5 GHz. For situations where C_x is not too large (roughly C_x values ≤ 10 fF), we can approximate the model frequency shift as $\triangle f = - b^* C_x$, independent of R_x, where b depends on microscope geometry. The model Q depends on C_x^2 as $Q = Q_{\max} - d(R_x)^* C_x^2$, where $Q_{\max}$ is the microscope Q with no sample present. For increasing R_x this slope increases in magnitude, roughly as $d(R_x) \sim R_x$. To summarize, in this small capacitance limit, the frequency shift image can be regarded as a capacitance image, and the Q image will contain contributions from both capacitance C_x and losses R_x. Similar results are obtained from a lumped element model in which the resonator is treated as a parallel RLC circuit.

In both models of the microscope we find in general that the minimum in Q versus R_x is always at the point where $\omega C_x R_x = 1$. Hence the sensitivity of our microscope to sample losses is determined in part by the value of the probe-sample capacitance. Other observations about the qualitative behavior of Δf and Q with sample properties have been discussed at length in prior work[9,16].

To summarize, the frequency shift image should be a map of probe-sample capacitance. Qualitatively, we expect C_x, to be large in a valley and small near a peak on the sample surface, as shown in Fig. 2. The capacitance between tip and sample can be calculated by assuming that the tip acts like a metallic sphere above a metallic infinite plane[20]. Naively, we expect the spatial resolution for capacitance variations to be on the order of the radius of the sphere.

Results: We find that the value of capacitance between tip and sample strongly depends on the geometry of the tip. We have used Pt-Ir alloy cut tips, as well as Pt-Ir alloy etched tips and W etched tips. All three tips have significantly different geometries. The W tip shows the largest Δf contrast as a function of height between tunneling and 2000 nm from the surface (Fig.3). For this tip we have seen a frequency shift slope $d(\Delta f)/dz|_{z\longrightarrow 0}$ contrast of roughly 0.3kHz/nm over a thin gold film deposited on a mica substrate. The etched Pt-Ir tip has a smaller contrast of 0.075kHz/nm, but still larger than a cut Pt-Ir tip, which has a contrast of 0.025kHz/nm, all on the gold on mica thin film (all three tips are compared in inset of Fig. 3). The largest frequency shift that we have seen is 800 kHz between tunneling height and 500 nm, over an oxidized Titanium sample with an etched W tip.

To quantitatively understand the frequency shift versus height data, we calculate capacitance for a given sphere radius and height above the sample starting from a typical tunneling height of 1 nm and extending to 2000 nm. The values are fed into the transmission line model[16,17], which calculates the frequency shift. The inset of Figure 3 shows the fit based on this model to the frequency shift versus height data for etched W and Pt tips over the Gold/mica sample. The sphere radius was used as the fitting parameter, and Fig. 3 shows that the W tip data fits well for a sphere of radius 27 μm, while the etched Pt tip fits with a sphere of 10 μm. These values for tip radius fits are comparable to those seen by other researchers[14,20] and give probe-sample capacitance on the order of 1 - 10 fF at tunneling heights. We find however, that the Pt-Ir cut tip is irregular and does not fit the sphere above the plane model.

Figure 4 shows simultaneously acquired images of STM topography and microwave properties on a $La_{0.67}Ca_{0.33}MnO_3$ CMR thin film. The STM topography clearly shows the granular structure of the film. The total height variation is about 175Å, and the smallest grain is about 285 Å on each side. The simultaneously acquired frequency shift data shows all the same granular features, with similar spatial resolution. Note that the frequency shift is more negative for the region between the grains, and less negative for regions near the top of the grains, as expected from Fig. 2.

Surprisingly, the Δf and Q image spatial resolution is just as good as STM topography. The contrast in Δf and Q come from the topography-following mode, where STM feedback is maintaining a constant tunnel current. As the tip goes into a valley on the surface, the microwave microscope will see an increase in capacitance between the tip and sample (Fig. 2) which will produce a more negative frequency shift. A stronger drop in Q is also seen due to the increase in C_x and possibly also due to a change in R_x, as proposed[21] for CMR thin films.

Figure 5 shows the data on the top of one grain of the $La_{0.67}Ca_{0.33}MnO_3$ film, shown in Fig. 4. This image is 492Å on each side and the overall STM topography is 96Å. The simultaneously acquired frequency shift image ranges from -105kHz to -110kHz and the Q image ranges from 348 to 357. The dark lines in the topography image are narrow dips about 8 to 10 unit cells deep. The Q and frequency shift images clearly show these dips as well. Figure 6 shows the line cut through the largest dip in Fig. 5, where the topography shows that this feature is 55Å deep, from the top of the grain. The frequency shift change is about 2 kHz and Q drops from 356.5 to 348.5 over this feature. One can regard Fig. 5 as a relatively flat region of the sample where the microscope shows a baseline frequency shift, Δf $\sim$ -105 kHz. When the tip moves into the 5.5 nm deep valley (Fig. 6), under STM constant-current mode, the microwave microscope shows an additional drop of $\sim$ 2 kHz in frequency shift, giving a slope, $d(\Delta f)/dz \sim 0.3$ kHz/nm, consistent with the results shown in Fig.3 for the frequency shift contrast near the surface.

This analysis suggests, that the STM constant-current mode is needed to see such sharp contrast in frequency shift, due to changes in capacitance. Hence, the spatial resolution will be dictated by the STM constant-current mode rather than the radius of curvature of the tip, as expected from the simple model of microwave microscope. Comparing the three line cuts through the feature (in Fig. 6), we clearly see that the spatial resolution for capacitance variations of the microwave microscope is comparable to STM, and is no worse than 25Å!

Noise ultimately limits our sensitivity to capacitance variations. There is noise in both the STM positioning system and the microwave microscope. At room temperature the

estimated position noise of the z piezo is 0.35Å and the position noise in x and y direction is 1.2Å. This translates to 0.0026 kHz of noise in Δf and 0.0083 in Q for a typical Pt-etch tip, so we conclude that the contribution to noise from the positioning system is negligible. We find that for the microwave microscope, sitting far away from the sample the jitter seen in the frequency shift signal is about 0.5kHz, and the variation in Q is about 0.1 out of 383. The lock-in time constant was 1 ms for these experiments.

Conclusions: We have demonstrated a novel microwave frequency scanning capacitance microscope with spatial resolution of no worse than 25Å. The microwave contrast depends strongly on the tip-sample capacitance. This capacitance depends strongly on the geometry of the tip. This scanning capacitance microwave microscope can serve as a high resolution platform for doing other kinds of measurements, such as local loss and local nonlinear properties[22] of interesting materials.

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FIGURES

FIG. 1. Schematic diagram of the STM-assisted scanning near field microwave microscope. A model of the probe-sample interaction is shown in the inset.

FIG. 2. Schematic of a)tip above flat region of the sample, b)tip in a valley and c)tip above a grain. The relative capacitance values between the tip and the sample are $C_{peak} < C_{flat} < C_{valley}$.

FIG. 3. Comparison of frequency shift vs. distance from tunneling height data for Pt-Ir etched (open circles) and W etched tips (open triangles) to the sphere above the plane model (solid lines). Experiments were performed at 7.37GHz. Inset plots the frequency shift versus log z to show the logarithmic distance scaling from the sphere above the plane capacitance model. The data are normalized to $\triangle$f (2000 nm) = 0 kHz. The inset also shows data for Pt-Ir cut tip (hollow squares), performed at 7.25 GHz. The sample is a thin Gold film on mica substrate.

FIG. 4. Simultaneously acquired a) Topography, b) Quality Factor and c) frequency shift image of $La_{0.67}Ca_{0.33}MnO_3$, thin film which is 1000 Å thick on $LaAlO_3$ substrate. The images are 6000 Å on each side. Data is taken att 272K with a Pt-Ir etch tip at 7.67 GHz.

FIG. 5. Images of the top of one grain of a $La_{0.67}Ca_{0.33}MnO_3$ a)STM topography b)Quality factor and c)frequency shift images. Image size is 492 Å on each side. The horizontal light and dark wide stripes on the topography image are due to temperature drift. Data is taken at 240K with a Pt-Ir etch tip at 7.67 GHz. The horizontal line cut in a) is shown in Fig. 6.

FIG. 6. Line cut of the data shown in Figure 5. It demonstrates a 25 Å spatial resolution for capacitance variations in the microwave response of the sample.